

# ***SUPERHARD COATINGS FOR ADVANCED VEHICLE SYSTEMS APPLICATIONS***

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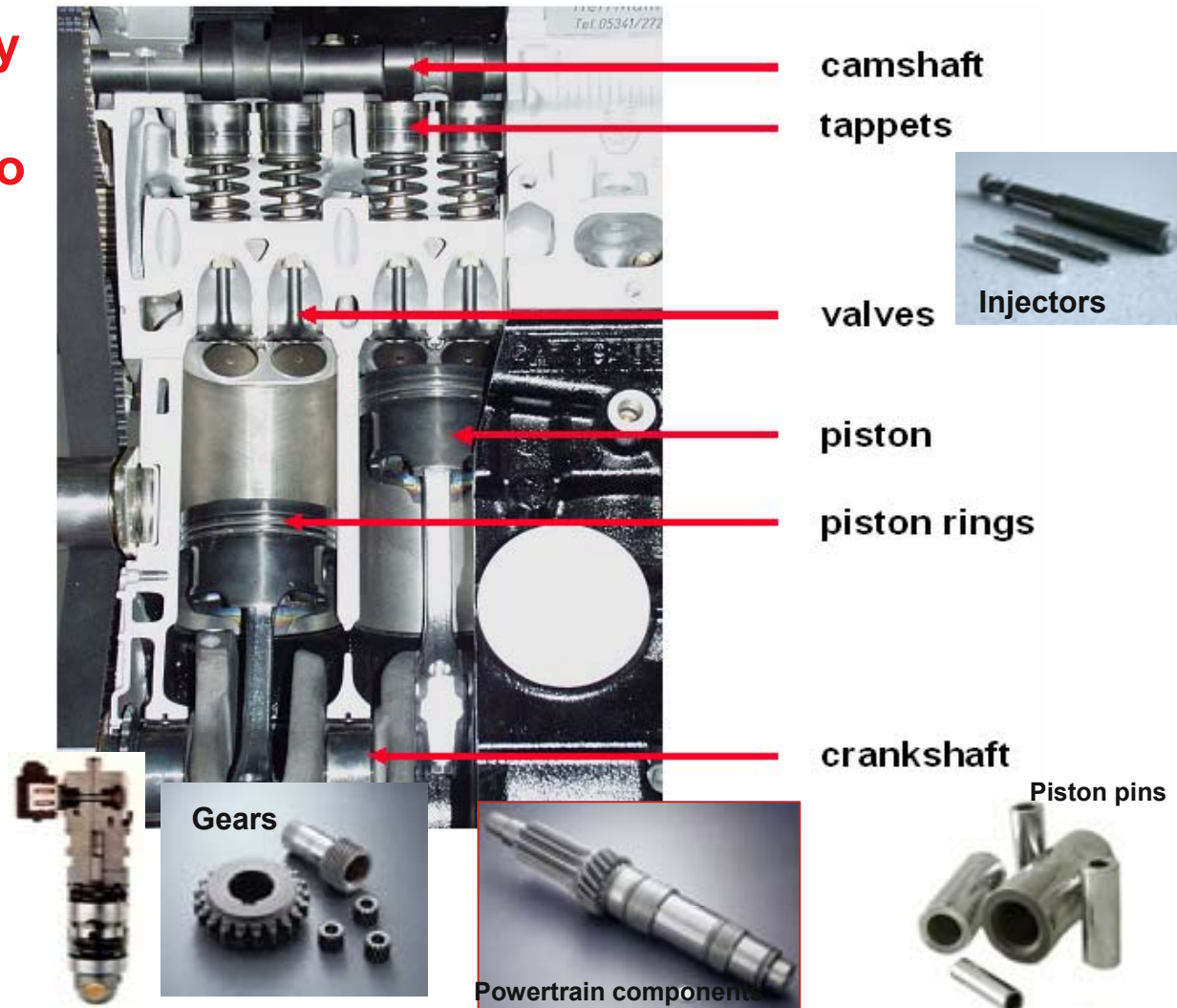
***April 19, 2006***



# Rationale and Goals

- Nearly 10% of fuel energy is consumed by friction in engines (which amounts to about one million barrels of oil/day)

- Design and develop new coatings to reduce friction
- Demonstrate their energy saving and wear reducing benefits
- Scale-up and transfer optimized technology to industry



# *Approach*

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- Design superhard coating composition for intended applications
- Develop a reliable deposition protocol for the production of superhard coatings
- Characterize structure and property
- Demonstrate performance
- Analyze test data and examine sliding surfaces
- Determine friction and wear mechanisms
- Integrate superhard coatings with laser texturing
- Prepare reports



## ***Industrial Collaborations***

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- **Burgess-Norton performing tests in fired engines (interested in licensing the technology)**
- **Eaton (a CRADA is underway)**
- **Caterpillar**
- **BorgWarner**
- **Westport**
- **Federal Mogul**
- **Coating Systems Manufacturers (Hauzer TechnoCoating, Ion Bond, CemeCon).**

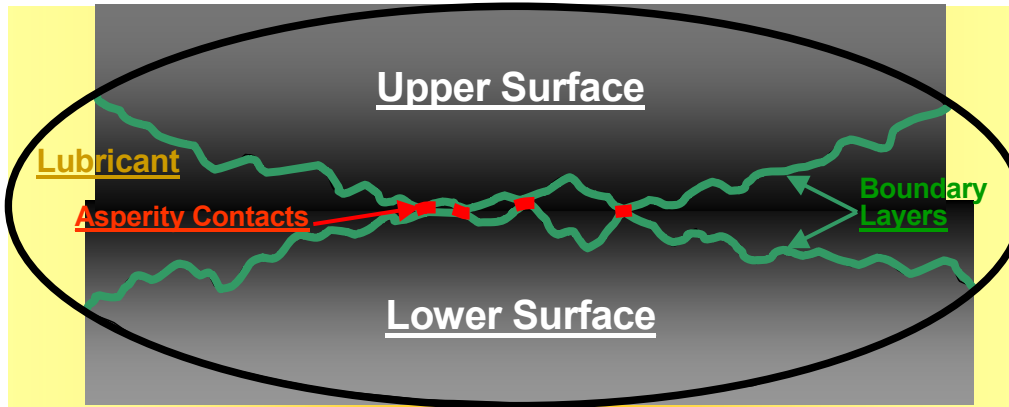


# *Technical Accomplishments*

- Successfully produced superhard coatings in a production-scale sputter ion plating system
- Demonstrated their superhard and low-friction nature
- Verified their extreme resistance to scuffing and wear
- Demonstrated their compatibility with and superior performance in EGR-contaminated oils
- Initiated surface analytical studies to understand lubrication mechanisms
- First measurements of residual stresses in thin coatings made using X-rays at the Advanced Photon Source
- Collaborated with an outside company to demonstrate its performance in piston pin applications



# Major Causes of Friction in Lubricated Contacts

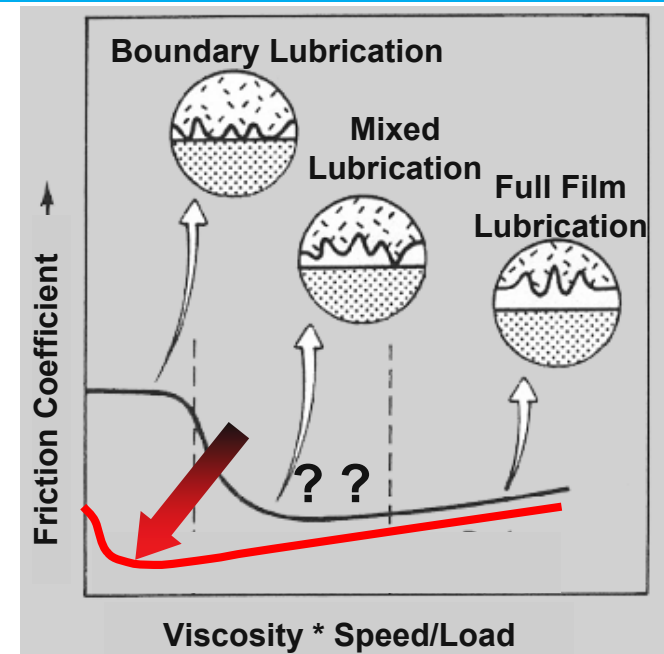


Schematic Illustration of Boundary Lubrication

In these components friction and wear result mainly from direct metal-to-metal contacts which often occur under high pressures, low sliding velocity and in low viscosity oils.

## Big Question ?:

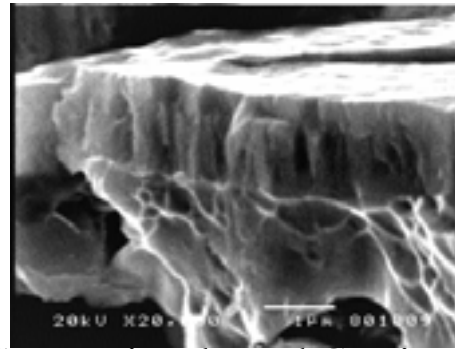
❑ Can we design coating systems that are not only superhard (for wear control) but also low-friction (for friction control) under severe boundary lubricated sliding conditions?



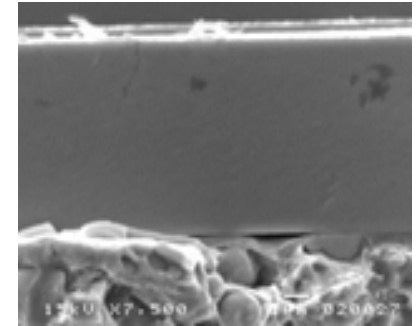
# Design and Synthesis of a Superhard, Nanocomposite Coating

Optimization of deposition process parameters is key to the development of these novel coatings providing superhardness, toughness and exceptional friction and wear properties.

SEM Cross-section Images

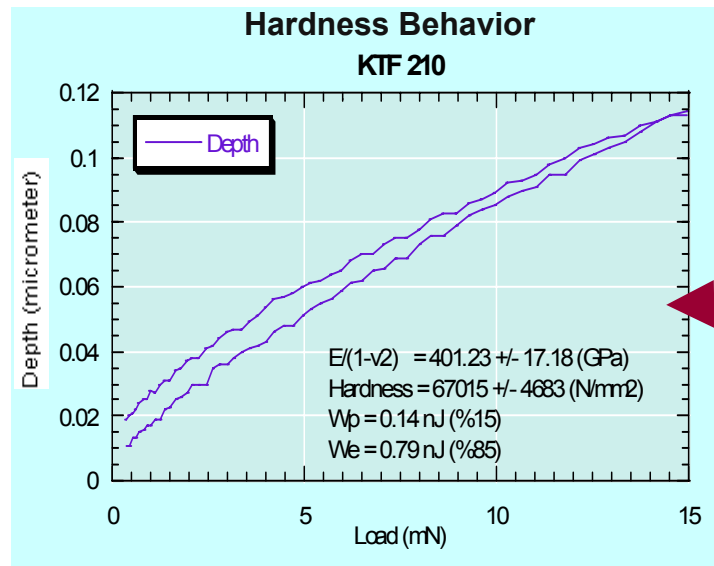
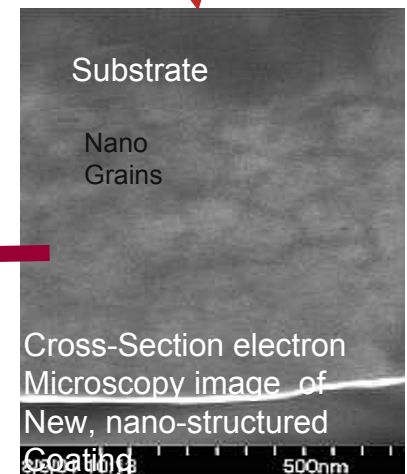


Conventional Hard Coatings



Superhard nano-composite coating

High-mag image



**Superhard:**  
**67GPa**

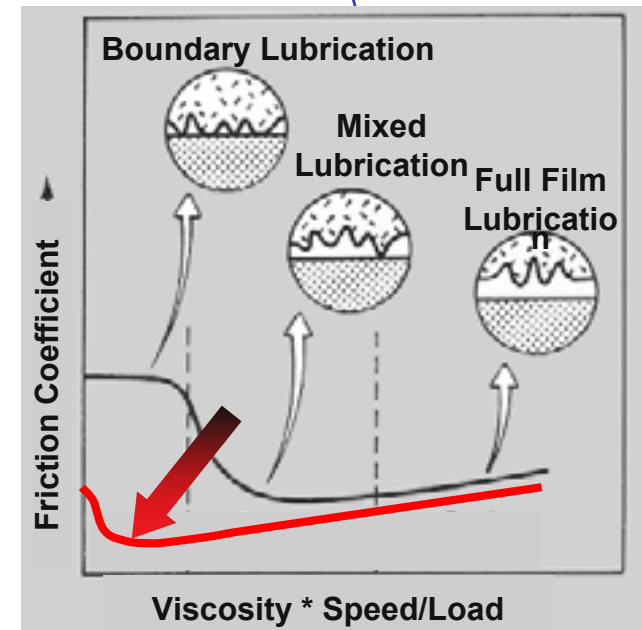
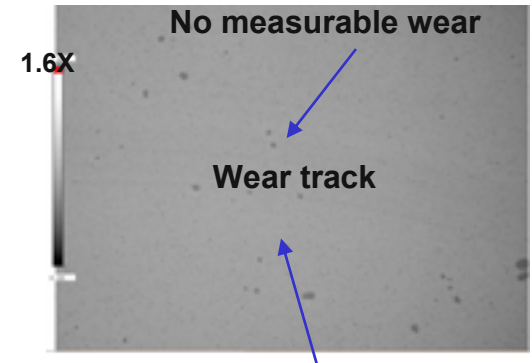
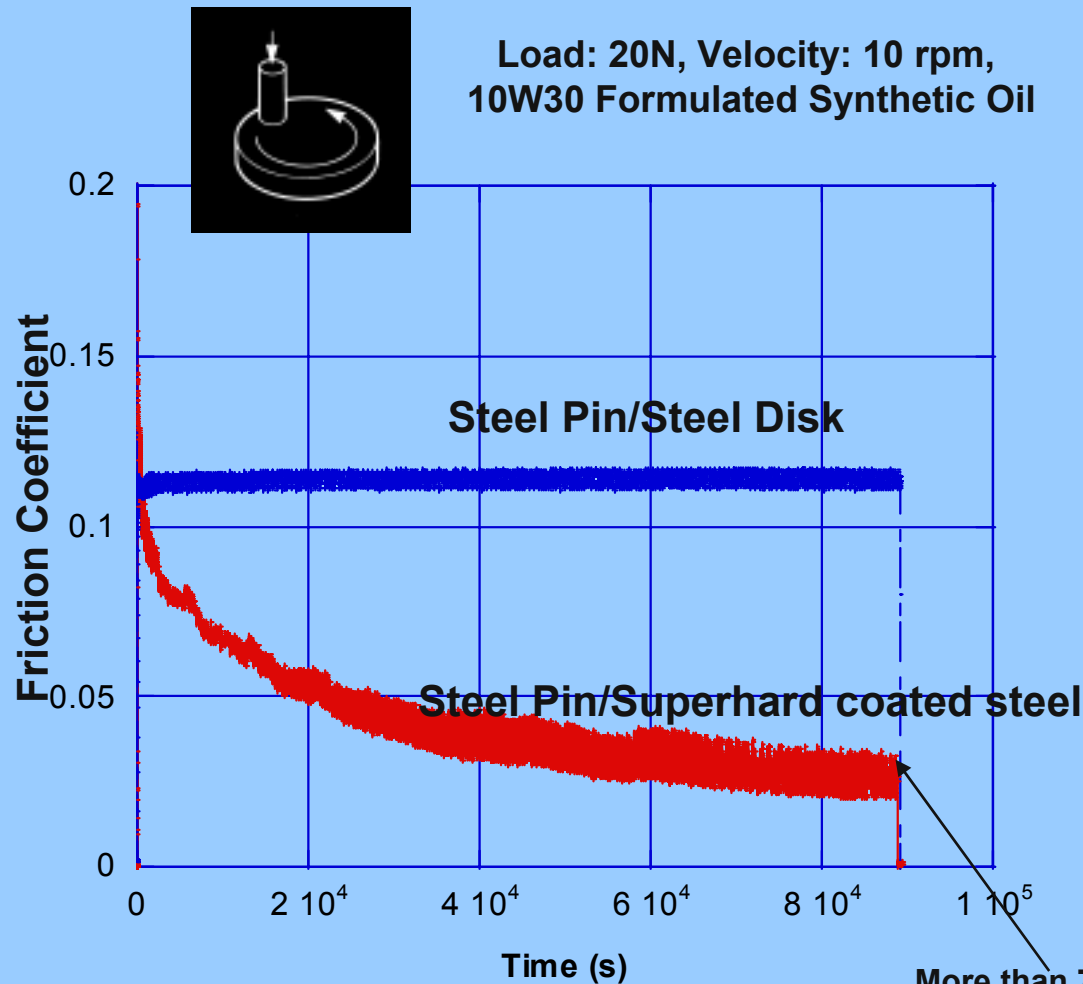


Sputter ion plating system

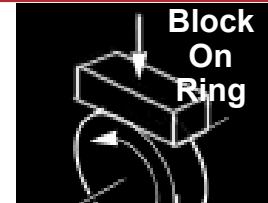
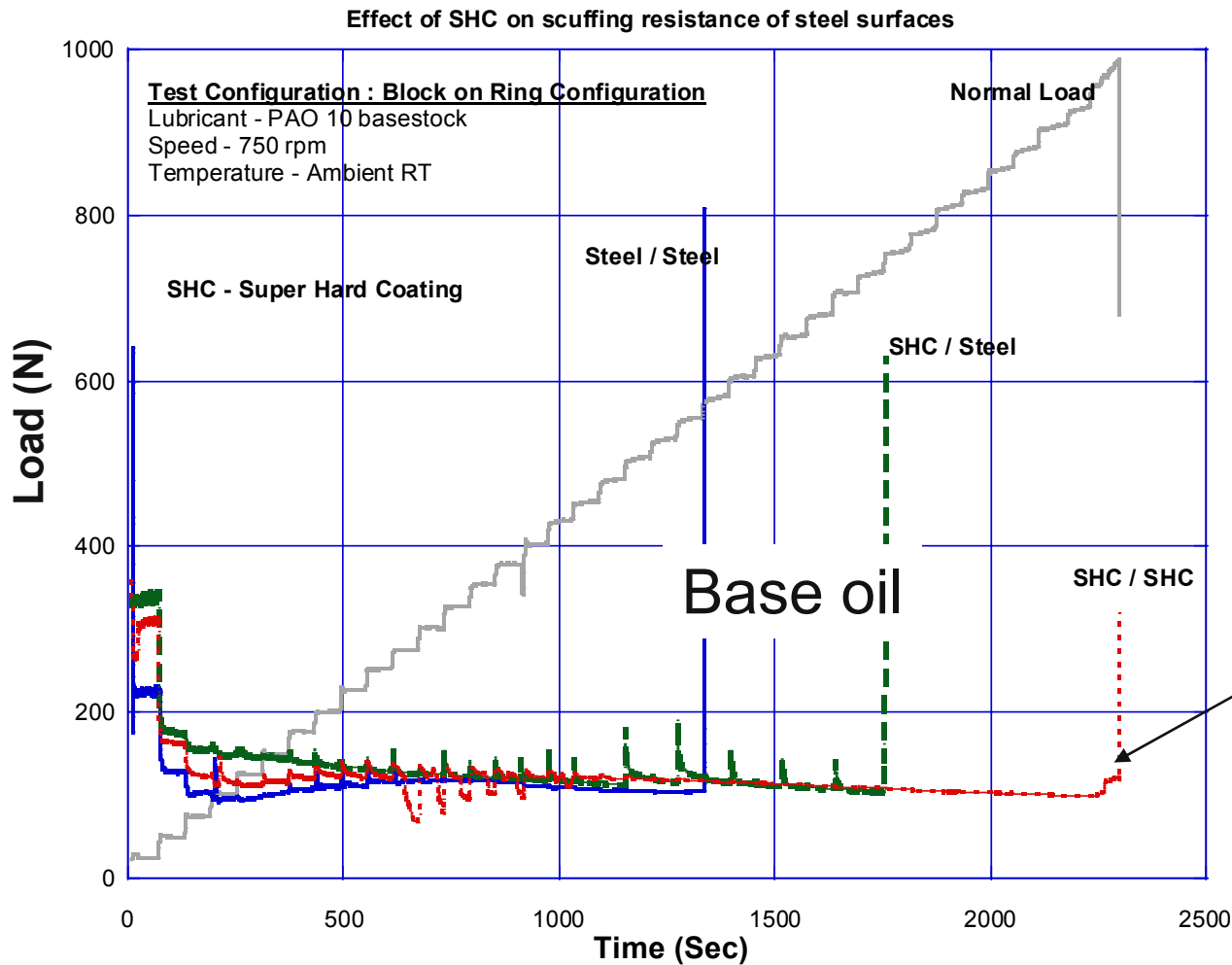
**Patent Pending**  
**Two more invention disclosures filed**



# Friction and Wear Performance Under Boundary Lubrication Regime



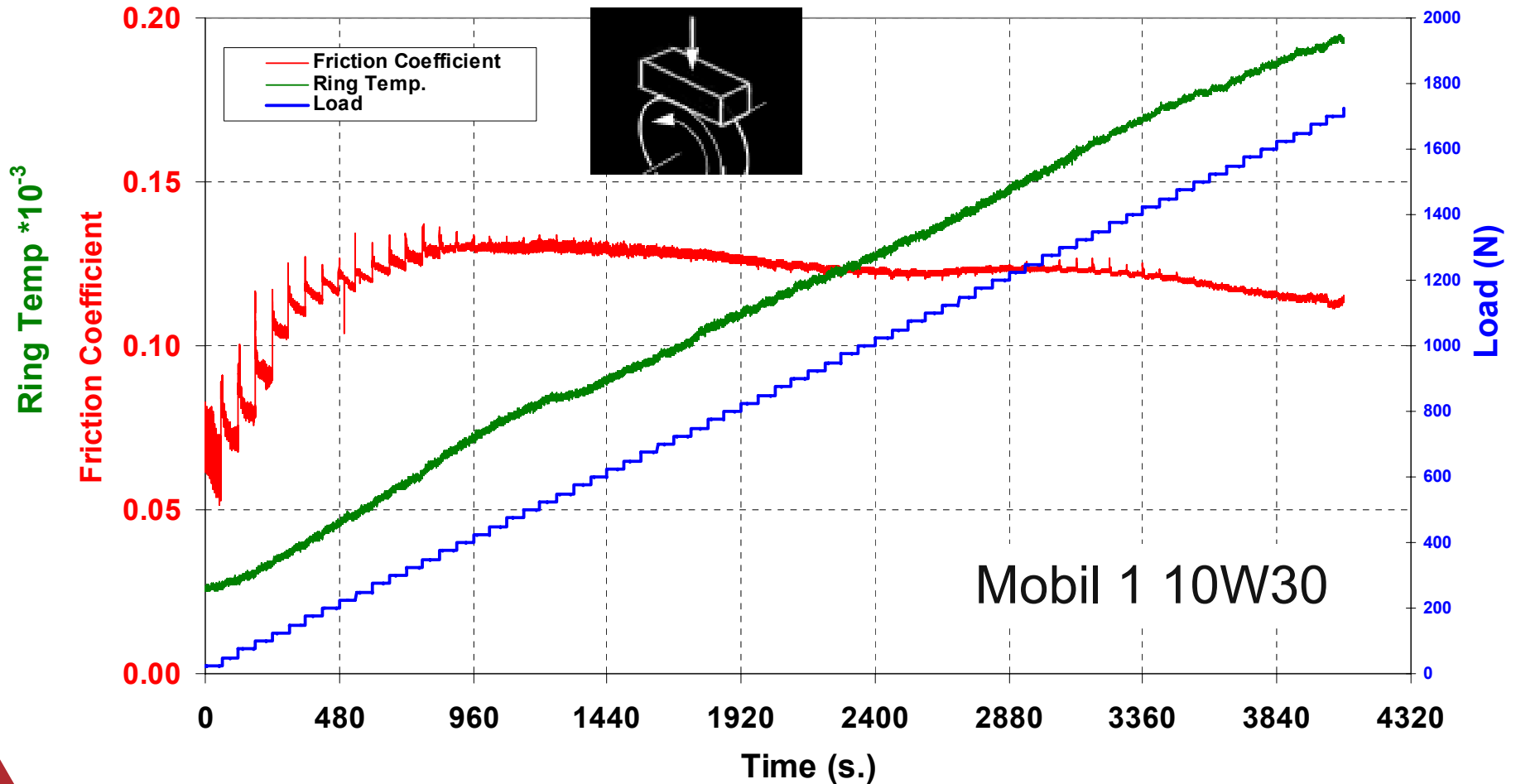
# Resistance of Superhard Coatings to Scuffing



Could not be scuffed.  
Test was terminated  
due to overheating  
of oil that produced  
heavy smoke.

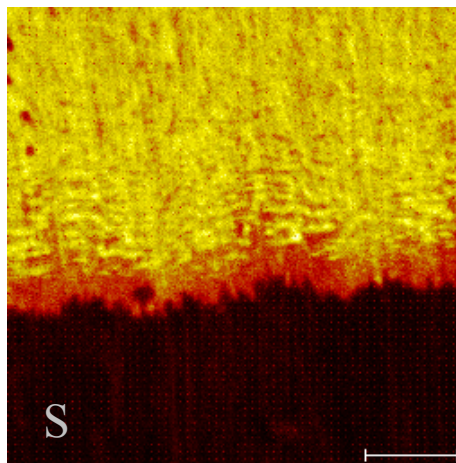
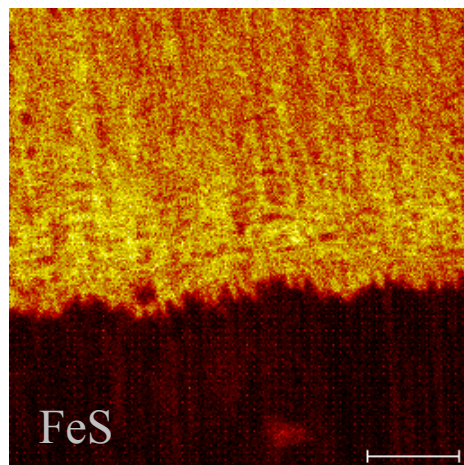
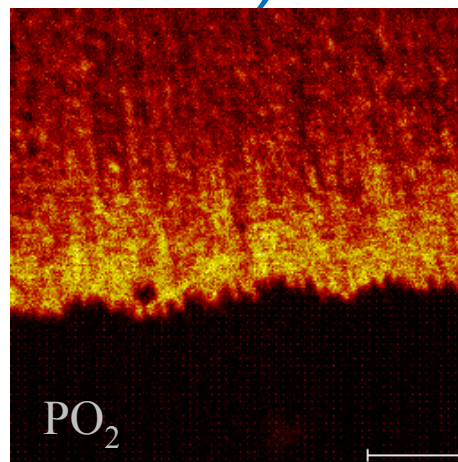
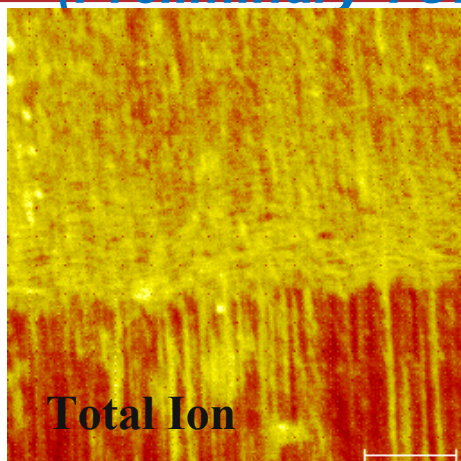


## Resistance of Superhard Coatings to Scuffing Cont'd

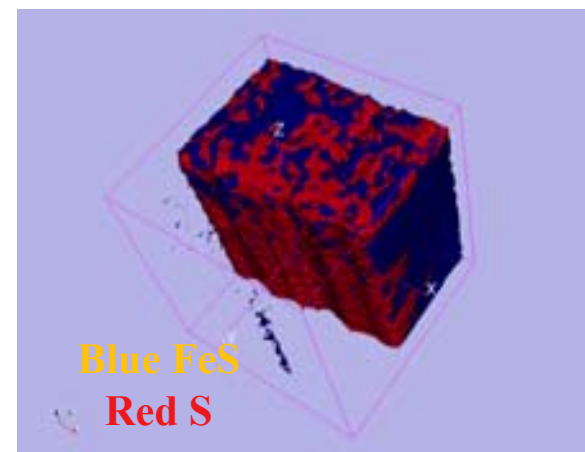


# Surface Analytical Studies

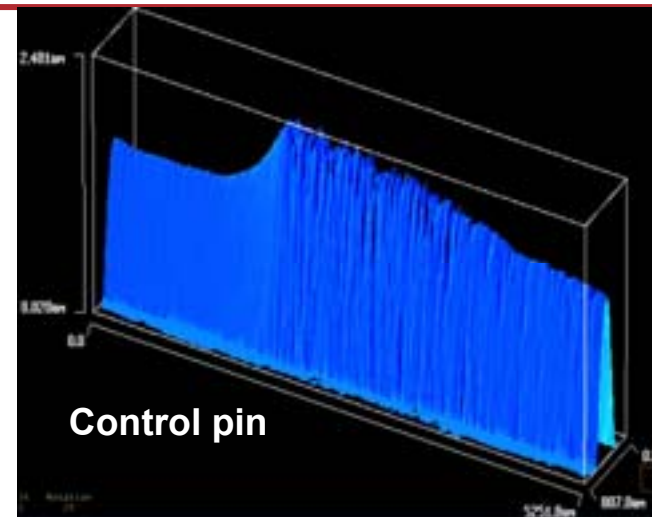
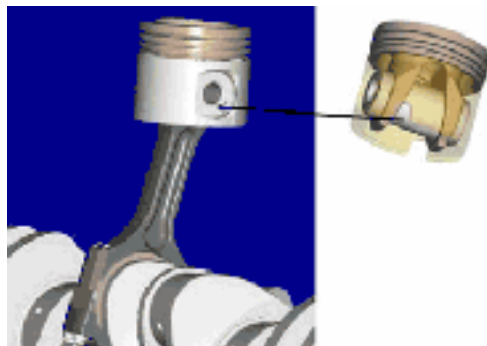
*(Preliminary TOF-SIMS Results)*



SHC coated ring vs.  
SHC coated block  
In Mobil 1 10W-30

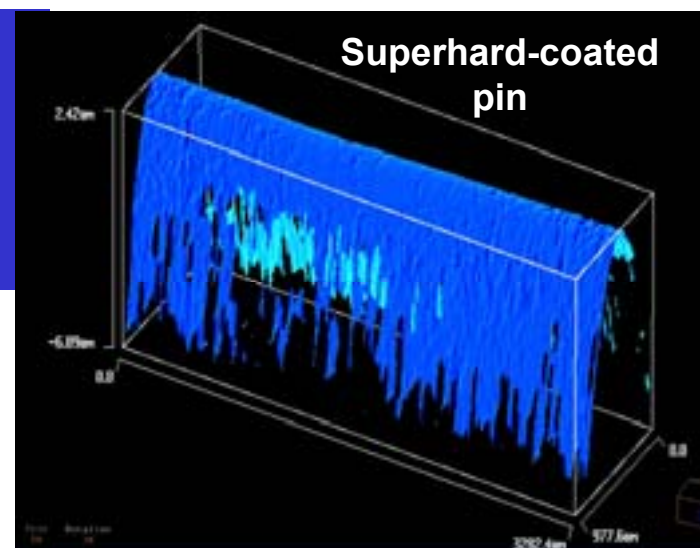


# Field Test Results: *Superhard Coatings on Piston Pins*



Tests were run at high speed (hence high temperature) for 100 hours in a V8 engine.

We had four coated pins in one bank of a V8 engine. The other 4 cylinders only had manganese phosphate coated pins (control pins).



# Summary

- A family of superhard coatings was successfully developed
- Their superior friction, wear, and scuffing performance was demonstrated
- Their resistance to EGR-contaminated diesel engine oils was confirmed
- Fundamental surface analytical studies and x-ray stress measurements were initiated
- Excellent results were obtained from limited field tests and more work is underway



## *Future Plans*

- Perform bench-top life and field tests
- Determine the effects of oil viscosity on friction and wear
- Perform more tests in EGR contaminated oils; explore corrosion/erosion related degradation
- Elucidate lubrication mechanism(s) using surface sensitive techniques (UIUC, ANL-APS).
- Elucidate the extent of internal stresses and correlate findings with performance and durability.
- Integrate superhard coatings with laser surface texturing to further enhance performance/durability
- Increase collaboration with industry, establish programs, transfer technology (potential customers; Caterpillar, Eaton, Federal Mogul, Burgess-Norton, Ford).



## ***Rationale: Residual Stress Measurements in Thin Coatings***

- Reduction of friction and wear in drive trains and engine components, and consequently, reduction in parasitic energy losses can result in >6% fuel savings
- Performance of low friction and wear coatings is strongly dependent on the residual stress profiles
- Currently, no technique is available to profile residual stresses in thin coatings

This is a new program in FY 06

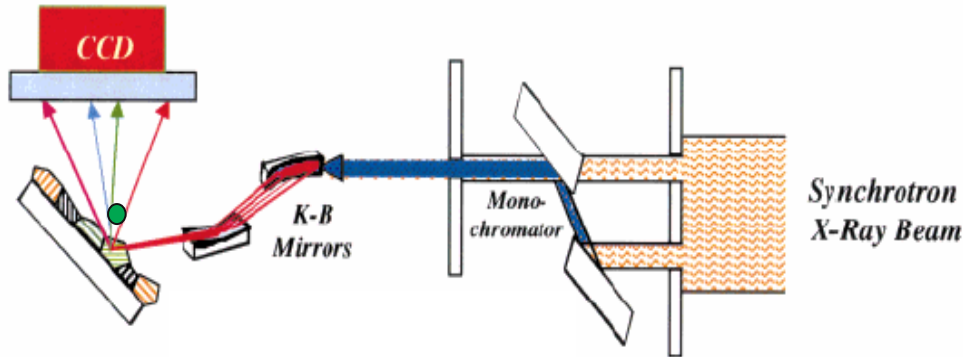


# Objectives

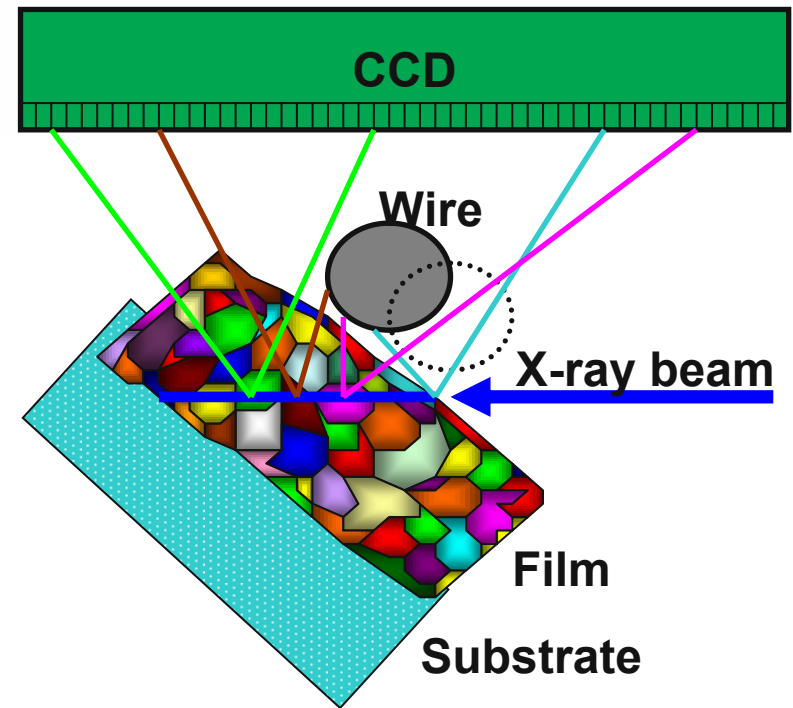
- Develop and refine high-energy X-ray technique(s) at the Advanced Photon Source (APS) to measure residual strains/stresses for super-hard, nanocrystalline low-friction coatings as a function of coating thicknesses
- Correlate residual stresses in coatings systems to processing technique and variables, material properties, and adhesion energies
- Compare experimentally measured residual stresses to calculated stresses from finite element modeling
- Develop an optimized coating processing protocol for a specific coating system and applications



# Differential Aperture X-Ray Microscopy (DAXM)

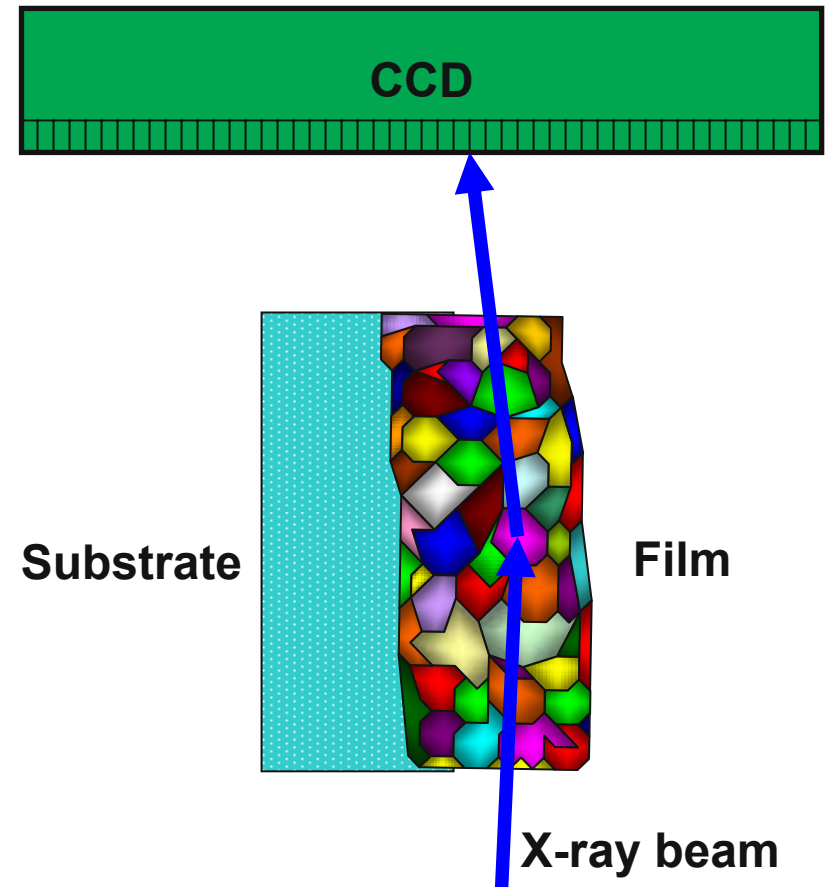


- Residual strains measured by comparing shift in diffraction spot positions from grains at specific coating depth with those of unconstrained grains
- X-ray absorbing wire allows depth profiling
- Depth resolution of  $\sim 0.5 \mu\text{m}$  is achievable

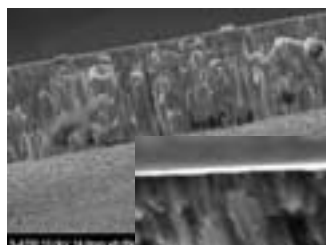


# Cross-Section Depth Resolved X-Ray Microscopy (CDRXM)

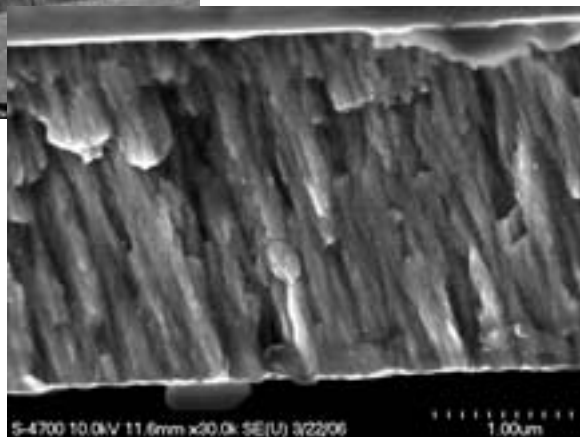
- Samples fractured to expose cross-section
- Depth of X-ray penetration dependent on energy ( $\sim 5\text{-}10\text{ }\mu\text{m}$ )
- Sample moved in steps of  $0.25\text{ }\mu\text{m}$  to scan surface of the film to substrate
- Invention report filed



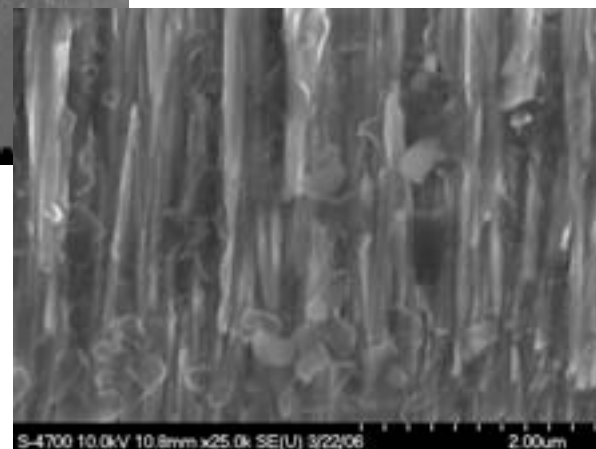
# Coating Systems Investigated



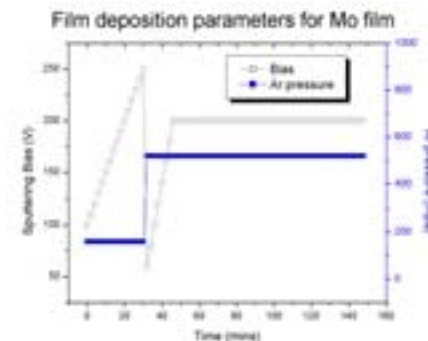
SHC on Si Substrate



Mo on Si Substrate



Sputter deposition  
200-300°C deposition temperature  
Ar pressure and sputtering bias  
Coating thicknesses ~ 3-5  $\mu\text{m}$

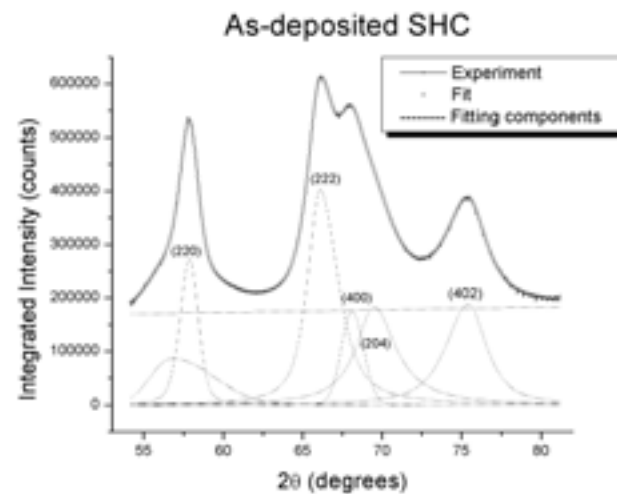
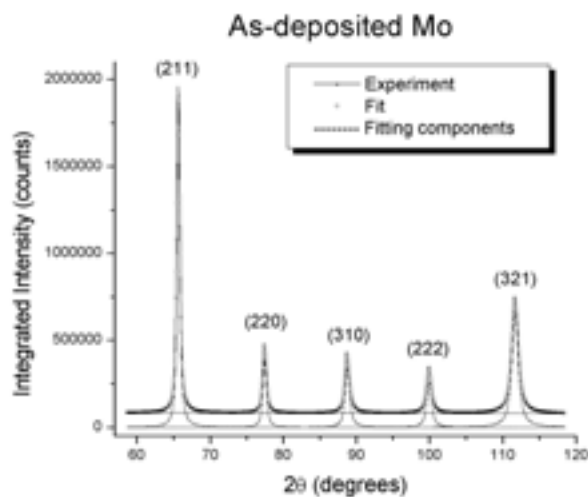
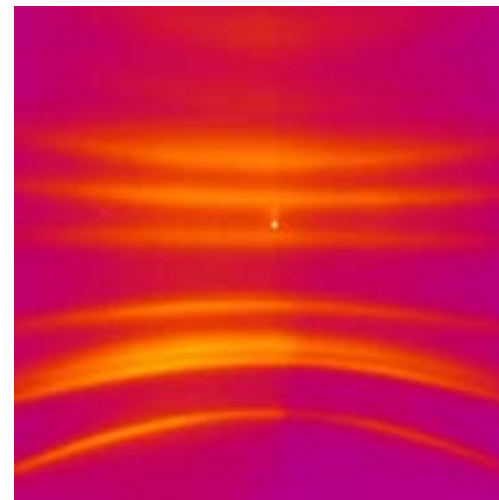
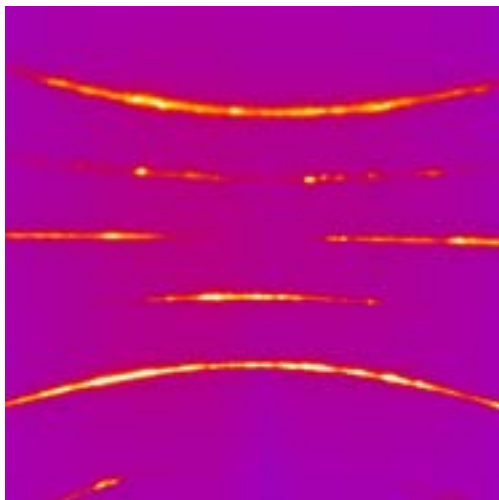


**Residual strains measured on as-fabricated and annealed (500 °C for 1 h) samples**

Samples fabricated by O. Eryilmaz (ANL)



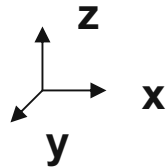
# Diffraction Data from CDRXM



# Residual strains measured by CDRXM: Mo coating

$\text{Si}_{\text{CTE}} = 2.6 \text{ ppm/K}$   
 $\text{Mo}_{\text{CTE}} = 4.8 \text{ ppm/K}$

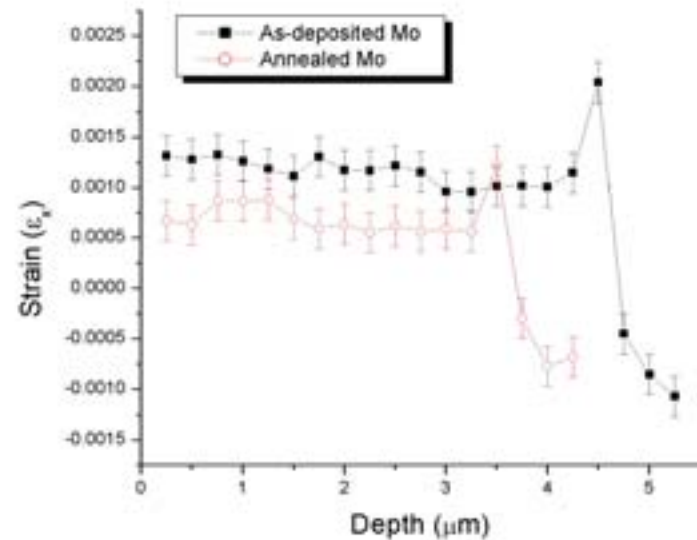
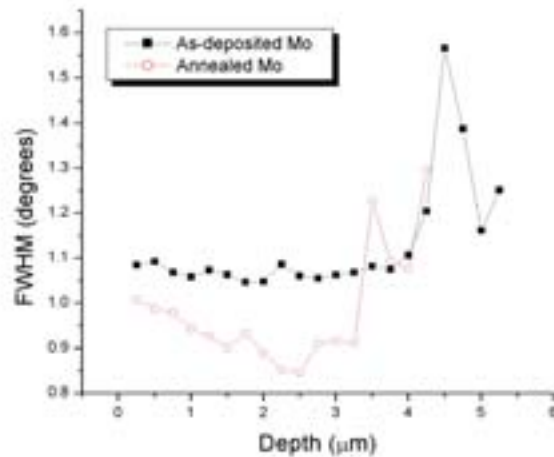
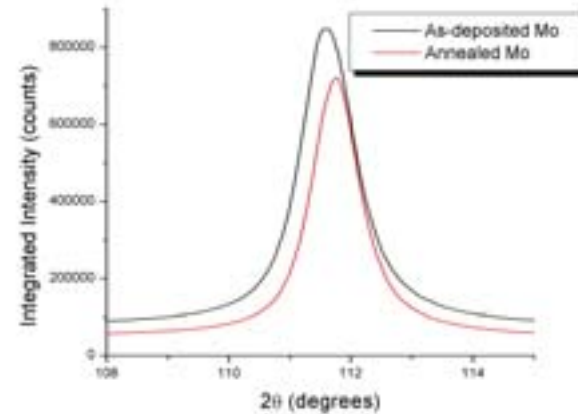
X-Rays



Mo →

Mo-large  
grains  
bond coat

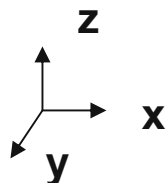
Si →



# Residual strains measured by CDRXM: SHC on Si substrate

$\text{Si}_{\text{CTE}} = 2.6 \text{ ppm/K}$   
 $\text{SHC}_{\text{CTE}} = ?$

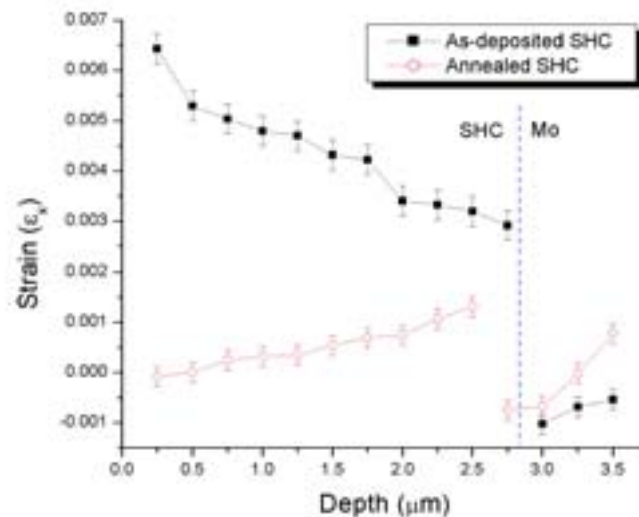
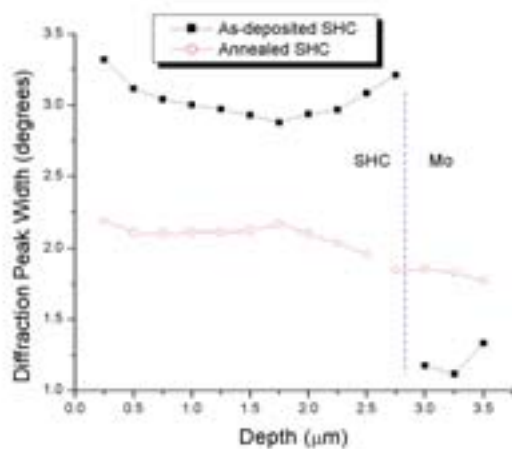
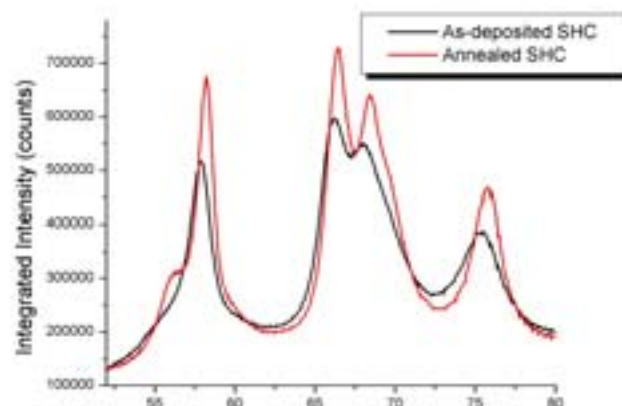
X-Rays



SHC →

Mo-large  
grains  
bond coat

Si →



# Summary

- For the first time, depth resolved residual strains in thin coating systems has been measured using CDRXM technique
- CDRXM technique, with sub-micron resolution, allows strain measurements even in the bond coat
- In-plane strains for Mo and SHC coatings are tensile and for the Mo bond coat they are compressive
- Magnitude of in-plane strains in SHC coating is significantly higher than in Mo coating and decrease from surface to the interface
- Annealing significantly decreases the in-plane strains for SHC, however, for Mo films, decrease in in-plane strains is relatively smaller



# *Future Plans*

- Compare residual strain measurements obtained from DAXM and CDRXM techniques (FY 06)
- Characterize coating adhesion properties using mechanical tests such as nano-indentation (FY 07)
- Compare strain measurements with FEM (FY 07)
- Measure residual strains/stresses in thin coatings fabricated under various processing conditions (FY 07)
- Correlate coating performance & adhesion with residual stresses & processing (FY 08)
- Develop a protocol for developing coatings with optimal properties (FY 08)
- Establish industrial collaborations to transfer the technology



# *Contributors*

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- O. Eryilmaz
- G. Chen
- B. Larson (ORNL)
- J. L. Routbort
- Weijin Liu

